

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of: Shulong Li
Serial Number: 09/718,807
Filed: November 22, 2000
For: **MULTI-LAYER COATING SYSTEM
FOR AIRBAG FABRICS**
Group Art Unit: 1771
Examiner: Singh, A.

**DECLARATION OF PRIOR INVENTION IN THE UNITED STATES OR IN
A NAFTA OR WTO MEMBER COUNTRY TO OVERCOME CITED
PATENT OR PUBLICATION (37 C.F.R. § 1.131)**

Box Non-Fee Amendment
Commissioner for Patents
Washington, D.C. 20231

PURPOSE OF DECLARATION

This declaration is to establish completion of the invention of this application in the United States at a date prior to June 7, 1999, that is the effective date of the prior art U.S. Pat. No. 6,239,046 to Veiga et al., that was cited by the examiner.

The person making this declaration is the inventor, Shulong Li.

FACTS AND DOCUMENTARY EVIDENCE

To establish the date of completion of the invention of this application and reduction to practice thereof, notebook entry reproductions are submitted as evidence and attached hereto within the Appendix A (with the actual dates blacked out as permitted by rule). These documents include blacked out dates that show conception and subsequent reduction to practice of this invention at least by a date prior to June 7, 1999.

DILIGENCE

As reduction to practice has been shown prior to the date of filing of the cited reference, a showing of due diligence is not necessary.

TIME OF PRESENTATION OF THE DECLARATION

This declaration is submitted prior to final rejection.

DECLARATION

As the person signing below as well as the person who invented the claimed invention and created the attached reproduced notebook entries:

I hereby declare that the date of conception and reduction to practice of this invention both occurred at least prior to June 6, 1999, a date prior to the filing date of the cited patent. Furthermore, I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these

statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

SIGNATURE

INVENTOR:

Full name of sole inventor: *Shulong Li*

Inventor's Signature: *[Signature]*

Date: *March 21, 2003*

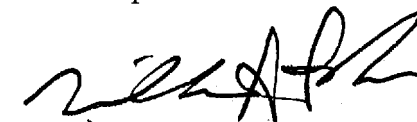
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CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to Commissioner for Patents, Washington, DC 20231, on March 21, 2003, along with a postcard receipt.



William S. Parks
Attorney for Applicant

APPENDIX A

Notebook Entries from Shulong Li's Records

A-Perm coating trial at Mascoe Systems

Formulation:

Sancure 12919	7400g
Micromid 632 hpl	7400g
DE-83R (70% disp.)	900g
Irgacure 1010 (30%)	400g
Netrosol 250 HXR	120g

Fabric: Autoliv Sacquard curtain air bag.

Trying to use the coatings to seal the fabric, especially the seam structure on the fabric to allow minimum leakage at 10-30 PSI air pressure.

Coating method:

Experimented, knife over roll

non-uniform coating

knife-over-air

non-uniform, ~~etc~~

2 knife coating

foam pad

We get 0.9 - 1.4 oz/yd² dry coating add-on on each side of the fabric. ~~leak~~ Air leakage at 10 PSI is ~~100~~ 60 - 115 SCFH

Date

Date

Signature

Witness

2 layer coat on Jacquard bag - A Perm.

Mix #1.	Ru 40 - 350	120 g
	Rhoplex E-358	60 g
	DE-83R (70%)	35 g
	Epi-Res 3519-W-50	8 g
	Irg. 1010 (30%)	8 g
	Nanguard 445 (30%)	5 g
	Tinuvin 292/1130	8 g
	Irgafos 168	5 g
	Aquamix 312	5 g
	Natrosol 250	1.7 g

Mix #2	BBE RR 1033 2150 g
	Rhoplex E-358 50 g
	Ampersce 5/1 120 g
	Aerofex M-3 5 g

Coat mix #1 on both sides of Jacquard bag first, and dried at 750°F for 2 min, then coat mix #2 on top of the first coating and dried at 750°F for 2 min.

Bag ^{is} ~~still~~ fairly soft.

Air leakage:	before aging	After aging
(end half)	3 scfh/9psi	7 scfh/9psi

25 Psi deployment, ~ 2 Psi at 4 sec.

Date

Date

Signature

Witness

A-7 Mascoe trial.

Base Coat :

Ru 40-350	6500 g
(lot #: 9809000020)	
DE-83R (70%)	400 g
Irganox 1010 (50%)	100 g
Nanguard 445 (30%)	130 g
Sunsperse pink	1 g
Natrosol 250 HXR	40 g

Top Coat :

AC 2000	3000 g
(Parachem)	
Amsperse FR 51	3700 g
Aerotex M-3	295 g
Nanguard 445 (30%)	30 g
Irganox 1010 (50%)	35 g
Sunsperse pink	4 g
Natrosol 250 HXR	82 g

Coated base coat on ¹⁰ Antolix curtain bags and other John Sellars bags at 1.5 y/min. ~~to~~ using profile 12 blade over foam pad and dried at 300°F. Then coat top coat using the same setting but dried at 340°F. Tested one ~~bag~~ Antolix bag after trial. ~8 SCFH / 9 PSI

add-on on John Sellars fabric :

0.78 oz/yd² base coat
0.68 oz/yd² top coat.

Date

Date

Signature

Witness

2 layer coating on woven air bags

We've found that a high tensile single layer polyurethane coating with a coating weight of 0.6 oz/yd^2 to 1.2 oz/yd^2 will seal up the Autoliv woven air bags.

Alternatively, a 2 pass 2 oz/yd^2 silicone coating will do a good job of sealing the air bag, especially at low air pressure. Polyurethane ~~seals~~ holds better at higher air pressure (15—30 psi).

Both silicone and polyurethane are expensive. A lower cost alternative is coat or dip coat the bag with a lower cost resin, such as polyacrylate, vinyl or other resins, then coat silicone or urethane on top of the base coat. In that way, much less silicone or urethane is needed, while overall performance will be equivalent to 100% silicone or 100% urethane coating. There, adhesion between the 2 layer will be critical. More work is needed to understand the effect of adhesion.

Date

Date

Signature

Witness

A-9 coating on side curtain air bags.

There are 2 surprises when I coated silicone on top of ~~conylat~~ polyacrylate, polyacrylate/urethane blend and polyurethane base coating:

- ① As long as the add-on of silicone is $\geq 0.7 \text{ oz/yd}^2$ or higher, the fabric passes FMVSS 302 test, doesn't matter there is any flame retardants in the base coat.
- ② Adhesion is fair — adhesion between silicone and polyurethane coating is good; adhesion between silicone and polyacrylate is fair; adhesion between polyurethane or polyacrylate and Nylon fabric is fair.

Air retention is reasonably good at 10 Psi.

When polyurethane is used as base coating, high pressure air leakage is low. Aging performance is good. — polyurethane is expensive!

When polyacrylate is used, however, low pressure air ^{leakage} ~~retention~~ is ~~good~~ ^{low}, high pressure air leakage is too high. Adhesion between silicone and polyacrylate is not very good. I have made some formulation change to improve that — continue on the next page

Date

Date

Signature

Witness

(Continue from the last page)

Adhesion promoter and crosslinker is used to improve adhesion and tensile strength of polyurethane.

Examples: polyfunctional epoxy
organofunctional silanes — epoxy silane
vinyl
melamine formaldehyde crosslinker

Expt. #1.	Rhoplex 32 NP	120 g
(base)	ECN 1400	10 g
(coat)	(Epoxy dispersion, CIBA)	
	Water	15 g
	Natrosol 250 HXR	1.8 g

Top coat: Dow Corning silicone 3625.

The base was coated using a round knife-over-foam pad, while top was coated using sharp knife over rubber pad. (Antolin small T-bag was used)

Adhesion between silicone and polyurethane is excellent.

Air retention: at 10 PSI, leakage is 3.5 SCFH

Repeat the coating on another T-bag. Air leakage

at 10 PSI is 2.5 SCFH. — They are surprisingly good.

Date

Date

Signature

Witness

Continue from the last page)

— Adhesion promoter and crosslinker is used to improve adhesion and tensile strength of polyamylate.

Examples: polyfunctional epoxy
 organofunctional silanes — epoxy silane vinyl
 melamine formaldehyde crosslinker

Expt. #1.	Rhoplex 32NP	120 g
(base)	ECN 1400	10 g
(coat)	(Epoxy dispersion, CIBA)	
	Water	15 g
	Nutrosol 250 HXR	1.8 g

Top coat: Dow Corning silicone 3625.

The base was coated using a round knife-over-foam pad, while top was coated using sharp knife over rubber pad. (Antolin small T-bag was used)

Adhesion between silicone and polyamylate is excellent.

Air retention: at 10 PSI, leakage is 3.5 SCFH

repeat the coating on another T-bag. Air leakage at 10 PSI is 2.5 SCFH. — They are surprisingly good.

Date

Date

Signature


Witness

Mascoe trial A-8.

Base coat:

Ru 40-350	4500 g
Rhoplex E-32NP	2100 g
Namguard 445 (30%)	100 g
Irg. 1010 (50%)	100 g
Natrosol 250	45 g

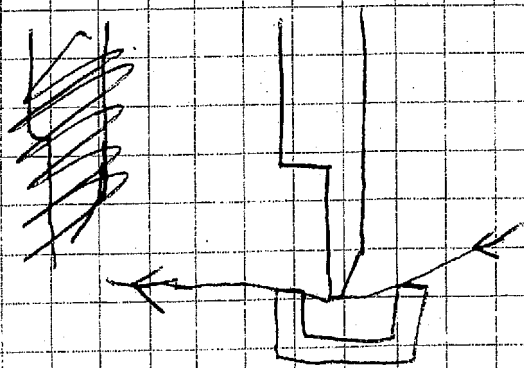
Top coat: Dow Corning 3625
(with pink pigment)

base coat was put on using knife over foam
pad. Drying temp 320°F. 1.5 y/min. 

Top coat was put on using knife over gap table.

1.5 y/min., 370°F drying temp

knife profile:



Date

Date

Signature

Witness

A-9 coating for curtain bag

Base coat:

Rhoplex E-32mp	100 g
Epi-Res 5520	8 g
Grilbond 2L-6	4 g
Natosol 250	1.2 g
Ing. 1010 (50%)	1.0 g

Top coat: Dow Corning 3625

A T-bag and a W bag were coated

Leakage of the T-bag: ~ 2.5 SCFH/10psi

Leakage of the W-bag: ~ 6 SCFH/10psi

W bag was reinforced at the corners w/
a thick layer of DC 3625 silicone.

When tested ~~on~~ on Charles's device for
pressure retention, after taken to 30psi

T-bag: 16.3 Psi/10 sec, 8 Psi/20 sec
1.9 Psi/40 sec.

W-bag: 11.3 Psi/10 sec. 3.7 Psi/20 sec.

They are not very good.

Date

Date

Signature

Witness

Effect of different amylate on the performance of A-9

Base coat.

Mix #1.	Rhoplex E-3273	120 g
	ECN 1400	10 g
	Water	15 g
	Natrosol 250	1.8 g

Mix #2	Rhoplex 3082	120 g
	ECN 1400	12 g
	Water	5 g
	Natrosol 250	1.8 g

Mix #3	Hycar 26345	120 g
	ECN 1400	10 g
	Water	15 g
	Natrosol 250	1.8 g

Top Coat Dow Corning 3625 (5/i)

Mix #3 gave the best pressure retention among the three. Both T-bags and W-bags were coated with above formulation. Mix #3 also gave the highest stiffness.

Date

Date

Signature

Witness

Robert A. [Signature]

A-9 Mascoe trial

Base coat mix =

Hycar 26345 (CBF Goodrich)	1000 g
Rhoplex E-32Np	4800 g
ECN 1400 (CIBA)	480 g
Epi-Res 5520	120 g
Irganox 1010 (50%)	100 g
water	720 g
Natroisul 250	65 g

Top coat : Dow Corning 3625
Silicone.

Base coat was put on using round blade
over a foam pad, and dried at 340°F.

Top coat was put on using shape knife
over a gap table and dried at 380°F.

16 Autoliv full size bags and ~ 20 Dobby
Woven Mercedes bags were coated.

Date

Signature

Witness

A-8/A-9 Polyurethane/amylate ratio,

Mix #1	Ru40-350	70 g
	Hycar 26731	27 g
	Aquamix 312	1.2 g
	Natrosol	1.2 g
Mix #2	Ru40-350	50 g
	Hycar 26731	44 g
	Aquamix 312	1.2 g
	Natrosol	1.2 g
Mix #3	Ru40-350	30 g
	Hycar 26731	62 g
	Aquamix 312	1.2 g
	Natrosol	1.2 g
Mix #4	Ru40-350	10 g
	Hycar 26731	80 g
	Aquamix 312	1.2 g
	Natrosol	1.2 g

All mixes were coated on T-bag and dried at 350°F for 2 min and top coated with Dow Corning 3625 silicone.

Mix #5 2 layer coating of DC 3625.

Date

Date

Signature

Witness

A-9 and A-8 expt. — Aging

Mix #1	Rhoplex E-32MP	110g
	Epi-Rez 5520	3.0g
	Water	20g
	Aquamix 3/2	2.5g
	Natrosol 250	1.7g
Mix #2	Rhoplex Hycar 2671	100g
	Epi-Rez 5520	25g
	Water	20g
	Aquamix 3/2	2.5g
	Natrosol	1.4g
Mix #3	Hycar 26731	100g
	Epi-Rez 5520	25g
	Water	20g
	Aquamix 3/2	2.5g
	Natrosol	1.3g
Mix #4	Ru 40-350	20g
	Epi-Rez 5520	20g
	Rhoplex E-32MP	60g
	Aquamix 3/2	2.0g
	Natrosol	2g
Mix #5	Ru 40-350	20g
	Epi-Rez 5520	15g
	Hycar 26731	65g
	Aquamix 3/2	2.0g

Date

Date

Signature

Witness

Continue

Continue from the last page.

Mix	Rn 40-350	10 g
#6	Epi-Rez 5520	25 g
	Rlycar 26731	65 g
	Aquamix 3/2	2.0 g
	Natrosol	2 g

Mix		
#7	Rhoplex E-32NP	110 g
	Epi-Rez 5520	30 g
	Water	70 g
	Aquamix 3/2	2.5 g
	Natrosol	1.7 g

Mix #8	Rhoplex E-32NP	110 g
	Epi-Rez 5520	30 g
	Water	110 g
	Aquamix 3/2	2.5 g
	Natrosol 250 HMR	1.5 g

Mix #9	Rn 40-350	30 g
	Epi-Rez 5520	20 g
	Rhoplex E-32NP	60 g
	Water	60 g
	Aquamix 3/2	2.0 g
	Natrosol	1.2 g

Mix #10	Rn 40-350	30 g
	Epi-Rez 5520	20 g
	Rhoplex E-32NP	60 g
	Water	100 g
	Aquamix 3/2	2.0 g
	Natrosol	1.6 g

Date

Date

Signature

Witness

Airbag coating using fluoropolymer resins.

Silicone coating has been popular due to its good aging stability, flexibility and heat resistance. ~~Other~~ Regular hydrocarbon

rubbers and elastomers would not have the same aging stability due to O_2 and water attack on the C-H, C-O, C-N ... bonds.

Fluoropolymers, on the other hand, has much ~~of~~ more stable C-F bond, and no, or few C-H bonds. ~~They are much more stable than~~ Fluoropolymer resins have much better aging stability than regular elastomers and rubber.

A good example will be the Kynar resin, PVDF homopolymer and copolymer. Most interesting ones are the copolymers of VDF with HFP and the blend of PVDF copolymer with acrylate.

Formulator:	Rhoplex E-32NP	50g
	Kynar 250/-20	25g
	DE-83R (70%)	10g
	Antiblaze Aceto NT F10	2g
	Water	20g
	Natrosol	0.8g

Date

Signature

Date

Witness

Autoluv T-bag coating

Mix #1.	Rhoplex E-32MP	25g
	Ru 40-350	70g
	Kynar 2501 (45%)	20g
	Water	15g
	Natrosal 250	1.5g

Mix #2 A-8 Mascoe trial retain
base coat.
Wacker LR 6289 top coat

Mix #3 A-8 Mascoe trial retain for basecoat
Wacker LR 6291 for topcoat

Mix #4	Rhoplex E-32MP	10g
	Ru 40-350	80g
	Dowlorning 61	15g
	Water	15g
	Natrosal 250	1.5g

Dowlorning 3625 was the ~~top~~ top coat for
mix #1 and #4

After 2 weeks aging in a 125°C oven, sample
#1 and #4 have far less color change (yellowing)
than #2 and #3 surprisingly. Small amount Kynar
and silicone latex can make that much difference

Date

Date

Signature

Witness